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OPTIMIZATION OF HEADSPACE SOLID-PHASE MICROEXTRACTION CONDITIONS FOR THE DETERMINATION OF VARIOUS INSECTICIDES IN TOMATOES AND CUCUMBER PULP

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EXTENDED ABSTRACT

The presence of pesticide residues in fruits and vegetables has aroused growing public concerns. Cases of intoxication due to consumption of contaminated agricultural food products happen from time to time. New analytical techniques that enable determination of pesticide residues in complex sample matrices with shorter turnaround time, improved sensitivity, and/or reliability are in constant demand. At present, most analytical methods in the literature involve extraction of pesticide residues from plant tissues by organic solvents, surfactants, supercritical fluids or solid-phase extractants followed by GC or HPLC determination. These extraction and cleanup procedures are usually tedious and time-consuming. Also, usage of environmental «unfriendly» organic solvents in most of these procedures imposes health hazardous to laboratory personnel and extra operational cost of waste treatment.

Solid phase microextraction (SPME) is a new and rapidly developing «solvent-less» solid-liquid extraction method. It involves extraction of analytes from the sample matrix (liquid or gaseous phase) onto an immobilized stationary phase. These extracted analytes may then be determined by GC via thermal desorption at the injector port or by HPLC via special injector interface. The advantages of SPME include true solvent-free extraction, high sensitivity, no need of cleanup procedures, and simple instrumentation. These make SPME an ideal tool for pesticide residue determination. In fact, there are already reports on the application of SPME for the sampling of organophosphorous pesticides in surface and groundwaters. However, literature on the feasibility of SPME determination of pesticide residues in agricultural products remains scare.

The aim of this work were to develop an efficient multi-residue method on the basis of headspace solid-phase microextraction (HS-SPME) and gas chromatography with FTD detection for the pre-concentration and chromatographic analysis of the selected organophosphorus pesticides, diazinon, fenitrothion, fenthion, parathion-ethyl, bromophos-methyl, bromophos-ethyl, and ethion in tomatoes and cucumbers pulp samples. The method was developed using spiked tomatoes and cucumbers pulp in a concentration range of 0.4-40μg/L. An increase in the extraction efficiency of OP insecticides was observed when the parameters affecting the HS-SPME process such as temperature, extraction time, salt additives, and stirring rate were optimized. Good linearity of compounds was observed in the tested concentration range. The limits of detection were between 0.04 and 0.2μg/L.

Key words: vegetable analysis, OP insecticides, HS-SPME, gas chromatography